FINAL TECHNICAL REPORT

Combustion Synthesis Technology Applied to *In-situ* Resource Utilization

by

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14. ABSTRACT
Solution combustion synthesis is different from solid and gas combustion in the presence of water. Fuels form natural resources were applied to the solution combustion of (SrAl2O4: Eu, Dy) compounds and properties of the products were studied. Technology to develop non-aggregating particles can be established with uniformly dispersed compounds.

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ABSTRACT

The present work(AOARD-05-4043) has been carried out for establishing a novel technology characterized with 'combustion synthesis of multi-functional compounds' and *in-situ* resource utilization'. The experimental approaches have mainly been focused on the following three items;
(1) to establish a new solution combustion synthesis technology,
(2) to produce nanometer-sized $SrAl_2O_4$ based long-duration luminescent materials, and
(3) to evaluate the nanometer-sized long-duration luminescent material potentials.

The interim report submitted in this January could cover the following problems under discussion and overview;
I.   "$SrAl_2O_4$ Structure Formation by Solution Combustion Synthesis" , which is for the partial fulfillment of the above items (1) and (2), and
II. "Technology for Fine Powder Solution Combustion Synthesis" for giving an effective solution to the item (2).

Solution combustion synthesis is different from others such as solid and gas reactions with respect to the existence of water. The merit/demerit of co-existing water situation (cluster formation or decomposition) can be made clear, and the morphology of products can be discussed under the consideration of heat passes during the combustion synthesis process. By applying the fuels of natural resources in solution combustion synthesis of ($SrAl_2O_4$: Eu, Dy) compounds, the properties of the products are investigated. The technology for obtaining non-aggregating particles can be established in order to form sheets with uniformly dispersed product compounds.

Through the assessment of nano-sized luminescent materials, the potential of nanometer-sized long-duration luminescent ones is discussed. By mixing these nanometer-sized particles with other nanometer-sized ones such as ZnO and Fe_3O_4, multi-functional materials can be expected as a next phase progress in practical applications.

For fulfilling the above problem II partially, a new technology has originally been proposed in the present work which can perform not only fine powders production but also highly efficient resource utilization.

This final technical report mainly focuses on the problem of "New Technology Proposal for Performing Nanometer-sized Materials" as a final output for solving the present three items including the summarizations of the previously reported problems I and II.

1. **Introduction**

Strontium-aluminum oxide ($SrAl_2O_4$) is one of typical matrices for long-duration luminescent materials, which are doped with Eu, Dy, Ce, etc. There have been several techniques in preparing such oxide powders; not only a solid reaction method but also a hydrothermal, plasma spray decomposition, sol-gel and combustion synthesis techniques. The long-duration luminescent materials have been applied in practical uses and admired as useful materials because of their energy saving lightening.

Nanometer-sized particles such as light-emitting silicon have recently been discussed as those achieving higher efficiency in the field of opto-science. In general, the luminescent intensity and afterglow lifetime of rare-earth elements doped $SrAl_2O_4$ are controlled with the relationship between the Sr-positions in the $SrAl_2O_4$ matrix and the doped Eu^{2+}, and with the distributions of Eu^{2+}, Dy^{3+}, etc. in the matrix, respectively. It was found that $SrAl_2O_4$ has a phase transition at 923K from a monoclinic phase to a hexagonal phase. The properties relating to the luminescent emission has been affected by the matrix phase, where the emission intensity was higher in the case of the hexagonal phase compared to the monoclinic phase. Since the priority in structure and/or size has been still under discussion because of unknown factors in luminescent efficiency, it is important to make clear the effect of nanometer-sized long-duration luminescent materials towards their higher functional properties.

Combustion synthesis does recently gather reputations as its effective synthesis technique, which can neglect such steps as washing, filtration, drying, etc. Combustion synthesis categorized with solid or gas reactions has almost been made clear without any consideration of *in-situ* pressure changes. Metal aluminates such as $SrAl_2O_4$, $CaAl_2O_4$, and $MgAl_2O_4$ can be synthesized by solution combustion synthesis, which consists of solution decomposition reactions and organic fuel combustion. Typical solution combustion synthesis has been carried out with urea($CO(NH_2)_2$), carbohydrazide($CO(N_2H_3)_2$), glycine($NH_2(CH_2)COOH$) were used as fuels. Recently, sucrose($C_{12}H_{22}O_{11}$) which has a larger molecule size has also been used as a fuel in order to increase distances between metal ions in the solution for the formation of fine products and the less agglomeration. Strontium nitrates($Sr(NO_3)_2$) decomposes into strontium oxide (SrO) at 925K, and aluminum nitrates($Al(NO_3)_3 \cdot 9H_2O$) decomposes into amorphous-Al_2O_3 at 443K. The difference between the decomposition temperature of $Sr(NO_3)_2$ and that of $Al(NO_3)_3 \cdot 9H_2O$ was more than 400K, therefore, the ratio of SrO and Al_2O_3 in the products may change. Since the combustion synthesis process proceeds with large temperature gradients in a short time, the difference of the decomposition temperature may not affect so much to the feature of products. The characteristics of solution combustion reactions, such as reducing power and generated gas amount, can be controlled by the selection of fuel, such as $CO(NH_2)_2$, $CO(N_2H_3)_2$, and $NH_2(CH_2)COOH$. The simple method to estimate total reducing power is introduced by propellant chemistry.

The basis of the combustion synthesis technique comes from the thermo-chemical concepts used in the field of propellants and explosives. The reaction releases the maximum energy when the reductive mixture follows its chemical formula. That is, when the element valences are balanced, irrespective of whether they are present in the oxidizer or the fuel components of the mixture. Thus, the method consists on

establishing a simple valence balance and the assumed valences are those presented by the elements in the usual products of the combustion reaction, which are CO_2, H_2O and N_2.   The extrapolation of this concept to combustion synthesis of ceramic oxides means that metals should also be considered as reducing elements with the valences they have in the corresponding oxides.   Experimentally, the chemical balance is used to calculate the appropriate amounts of the selected starting materials (i.e. the cationic precursors and the fuel) following its chemical formula designed for expected product composition.   This concept is particularly useful when thermodynamic calculations are difficult to carry out for lack of the relevant parameters and it has been shown that there is a direct correlation between the results derived from the valence balance and those based on heat of formation or bond energies.   When both the precursor salts and the fuel are water soluble, a good homogenization can be achieved in the solution.

In the present work, the utilization of natural minerals in solution as the reactants for combustion synthesis is evaluated and applied to the production of long-duration luminescent nanometer-sized particles.   With a newly controlled process proposed in the present work as shown in Fig. 1, the possibility of the solution combustion synthesis potential is discussed in fundamental and practical approaches.
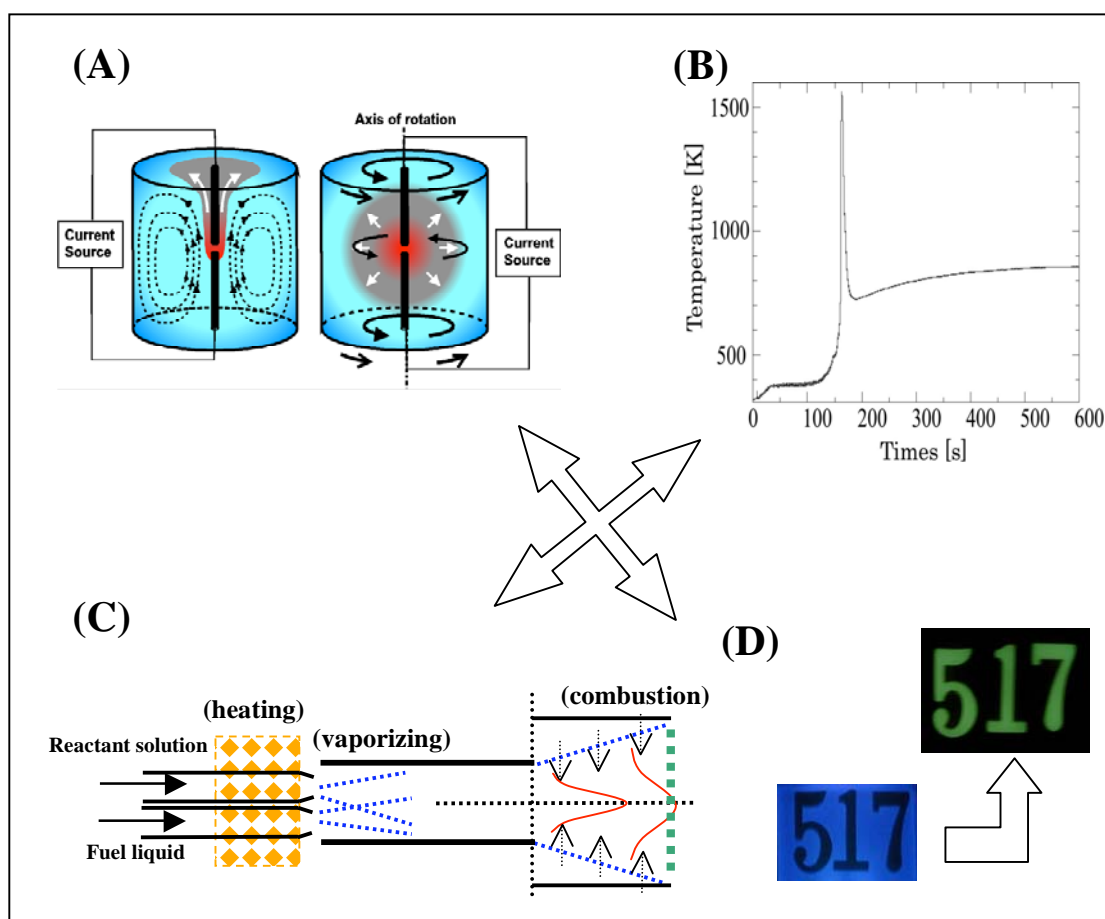

Fig. 1   Technical combinations proposed in the present work

(A): Convection control with wall-rotations, (B): Solution combustion synthesis,
(C): New combustion system designed with liquid-mixtures,
(D): Performance of long-duration luminescent materials

2. Classification of combustion synthesis technologies

An early application of combustion synthesis was in 'thermite' reactions of metal oxide and aluminum powders which can produce metal elements and/or alloys with aluminum oxides. The heat generated by their exothermic reactions was sufficient even for applications to welding, process heat treatment, etc. The research efforts have been able to propose productions of over 500 compounds by combustion synthesis. Some applications of combustion synthesis can be classified as follows;

- Abrasives, cutting tools and polishing powders, e.g. TiC, cemented carbides.
- Resistive heating elements, e.g. $MoSi_2$.
- Shape memory alloys, e.g. TiNi.
- High temperature inter-metallic compounds, e.g. Ni-Al.
- Steel processing additives, e.g. ferro-nitrides.
- Electrodes for electrolysis of corrosive media, e.g. TiN, TiB_2.
- Coatings for containment of liquid metals and corrosive media, e.g. products of Al and Fe oxide thermite reactions.
- Powders for further ceramic processing, e.g. Si_3N_4.
- Thin films and coatings, e.g. TiB_2.
- Functionally graded materials, e.g. TiC-Ni.
- Composite materials, e.g. $TiC+Al_2O_3$, $TiC+Al_2O_3+Al$.
- Functional materials, e.g. $BaTiO_3$, $YBa_2Cu_3O_7$.

Compared with conventional processes, main advantages of combustion synthesis are;

- The generation of a high reaction temperature which can volatilize low boiling point impurities and, therefore, result in higher purity products;
- The simple exothermic nature of the SHS reaction avoids the need for expensive processing facilities and equipment;
- The short reaction time results in low operating and processing costs;
- The high thermal gradients and rapid cooling rates can give rise to new non-equilibrium or metastable phases;
- Inorganic materials can be synthesized and consolidated into a final product in one step by utilizing the chemical energy of the reactants.

These advantages have intrigued researchers to become more active in exploring the combustion synthesis approach of new and improved materials with specialized mechanical, electrical, optical and chemical properties. However, there has also been some considerable research devoted to improvement of the final product quality, particularly with respect to reducing porosity.

There are a number of reaction parameters which affect combustion synthesis reactions, e.g. reactant particle size, stoichiometry (including the use of diluents or inert reactants), green density, thermal conductivity, ignition temperature, heat loss and, therefore, combustion temperature, heating and cooling rates and physical conditions of reactants (solid, liquid, gas). Many of these parameters are interdependent and have a significant effect on the final product morphology and properties. Establishing the optimum reaction parameters for synthesizing a material is based on obtaining a fundamental understanding of the controlling reaction mechanisms in each combustion reaction system. This has been one of the most active research areas for combustion synthesis. Combustion synthesis has emerged as an important technique for the synthesis and processing of advanced ceramics, catalysts, composites alloys

inter-metallic, and nanometer-sized materials. Depending on selections of characteristic reactants, the combustion synthesis technology can be classified as shown in Fig. 2, where the reactant elements or compounds are varied in solid, liquid or gas phase and each nature of exothermic reaction characterizes combustion propagation and system temperature controlled processes.
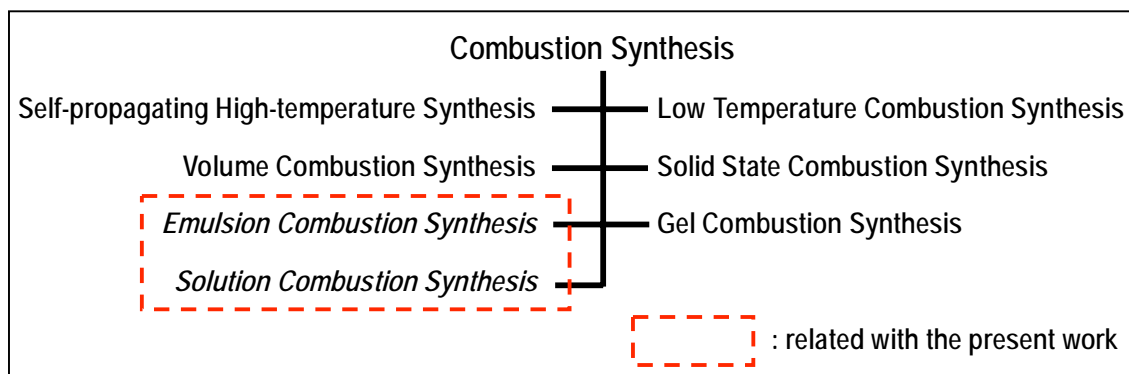

Fig. 2   Classification of combustion synthesis

Solution combustion synthesis of preparing oxide materials is a fairly recent development compared to solid-state combustion synthesis or self-propagating high-temperature synthesis technologies. Today, solution combustion synthesis is being used all over the world to prepare oxide materials for variety of applications. In the case of dry processing, product powders form agglomerates of non-uniform size and distribution. This could lead to poor mixing of the multi-component reactants and to packing density variations, which may result in inhomogeneous structures in the synthesized products. However, in wet processing, the particles are dispersed in a liquid and are free to move in relation to each other so better mixing is achieved. It is possible to prepare oxide materials with desired composition, structure, etc. by solution combustion synthesis. The oxide materials prepared and investigated during the review period have been along with the fuels used and their applications. The knowledge of the adiabatic temperature and the physical state of each reactant can give the first basis for the determination of the synthesis mechanisms, and the chemical and physical processes are identified, which affect the structure formation during the combustion synthesis:
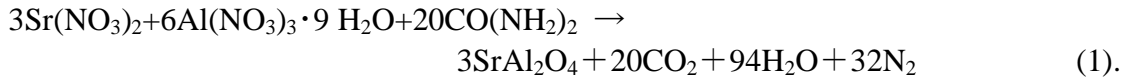
1. Heat transfer from the reacted to the non-reacting zone;
2. Phase transition of solid reactants;
3. Melting of reactants;
4. Spreading of a molten phase;
5. Gasification of volatile impurities and reactants;
6. Chemical reaction with initial product formation;
7. Melting of intermediate products;
8. Melt crystallization upon cooling;
9. Crystal growth;
10. Phase transition in solid products during cooling;
11. Ordering of the crystal structure.

The steps from 1 to 7 occur in the heating part of the combustion, and the steps ranging
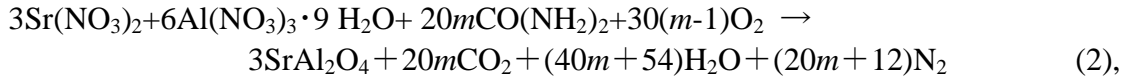
from 8 to 11 for the cooling part.   Therefore, the control of the combustion propagation process is much important for its performance.

3. $SrAl_2O_4$ structure formation by solution combustion synthesis

When using the urea as the fuel, its combustion reaction propagates much milder compared to other fuels.   The reaction of solution combustion synthesis using $CO(NH_2)_2$ is shown in the following equation (1),

$$3Sr(NO_3)_2+6Al(NO_3)_3 \cdot 9\ H_2O+20CO(NH_2)_2 \ \rightarrow$$
$$3SrAl_2O_4＋20CO_2＋94H_2O＋32N_2 \qquad (1).$$

The heat of reaction calculated for one mole of $SrAl_2O_4$ from the equation (1) is 3100 kJ/mol, and the adiabatic temperature is 1090K.   In the cases of $CO(N_2H_3)_2$ and $NH_2(CH_2)COOH$, those are calculated as 5300kJ/mol and 3211kJ/mol, respectively. To gain some excess of heat, the ratio of urea can be added to the equation (1), which is shown in the equation (2),

$$3Sr(NO_3)_2+6Al(NO_3)_3 \cdot 9\ H_2O+ 20mCO(NH_2)_2+30(m\text{-}1)O_2 \ \rightarrow$$
$$3SrAl_2O_4＋20mCO_2＋(40m＋54)H_2O＋(20m＋12)N_2 \qquad (2),$$

where the m shows the urea excess. In the case of $m=1.5$ and 2.0, their heats of reaction are calculated as 5237 and 7313kJ/mol, respectively.   Those adiabatic temperatures are determined as 1300K and 1410K, respectively.

The typical temperature change during combustion synthesis reaction is shown in Fig. 3.   The maximum temperatures were more than 1270K, which appeared from 370K in a few seconds, and were almost the same independent of urea amounts and furnace temperatures.   Therefore, in the reactions of combustion synthesis, $Sr(NO_3)_2$ and $Al(NO_3)_3 \cdot 9H_2O$ can react simultaneously to synthesize $SrAl_2O_4$ as shown in the equation (1). The present results i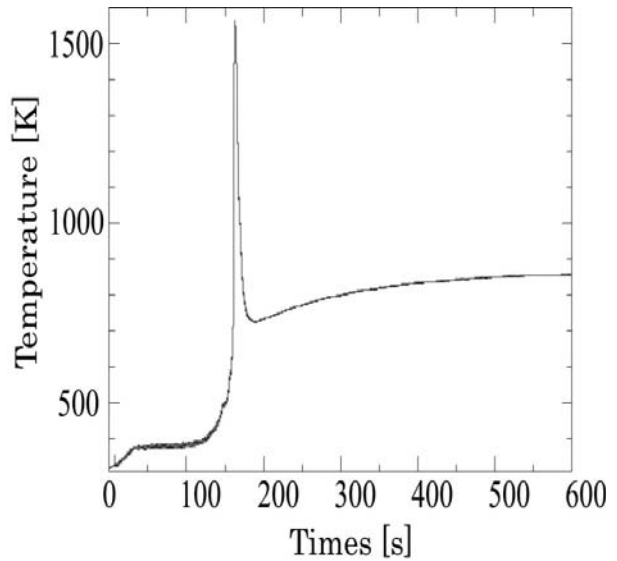ndicate that the combustion synthesis reaction can be performed above 870K of the furnace temperature, which means that the present solution combustion synthesis is initiated at least more than 870K as the pre-heating temperature.

Fig. 3   Process temperature change
(furnace temperature: 870K)

Composition ratios of products varied with the temperatures of pre-heatings and urea ratios are shown in Figs. 4 and 5.   Since the peaks of monoclinic-$SrAl_2O_4$ (α-SA), hexagonal-$SrAl_2O_4$ (β-SA), $Sr_3Al_2O_6(S_3A)$ and $Sr_2Al_4O_7(SA_2)$ were overlapped,   the identification was difficult. Therefore, the composition ratios were calculated from

simultaneous equations of several peaks heights.

With increasing the pre-heating temperatures from 870 to 1270K, β-SA over the whole temperature range, and the ratio of α-SA became the maximum at 1070K. As the furnace temperature increases, the ratio of S_3A increased and that of SA_2 decreased.

These results can show that a part of $Sr(NO_3)_2$ does not react and the ratio of SrO is insufficient at low temperatures. Non-reacted part of $Sr(NO_3)_2$ is decomposed into SrO with the increase of furnace temperatures, because the reaction temperature between $Sr(NO_3)_2$ and $CO(NH_2)_2$ would be around 870K. The ratio of product composition changed from the region of $SA+SA_2$, through that of SA, to that of $SA+S_3A$ because insufficient SrO decreased with increasing temperatures of the furnace.

The SEM images are shown in Fig. 6. Particle sizes became finer with the increase of pre-heating temperature. It is also suggested that the reactivity of samples should be improved as increasing furnace temperature. The ratio of α-SA and β-SA was maximum and the ratio of S_3A and SA_2 was minimum at 1.5 times of $CO(NH_2)_2$ stoichiometric amount. At the stoichiometric ratio of $CO(NH_2)_2$, a part of $CO(NH_2)_2$ would be evaporated and the reaction system was in the state of insufficient of $CO(NH_2)_2$. When 1.5 times stoichiometric ratio of urea, the ratio of nitrates and fuel was considered to close to theoretical ratio, and when 2.0 times stoichiometric ratio of $CO(NH_2)_2$, the ratio of fuel might be too excess. These results suggested that phase composition of strontium aluminates can be controlled by changing combustion synthesis condition, such as furnace temperatures and urea amount.
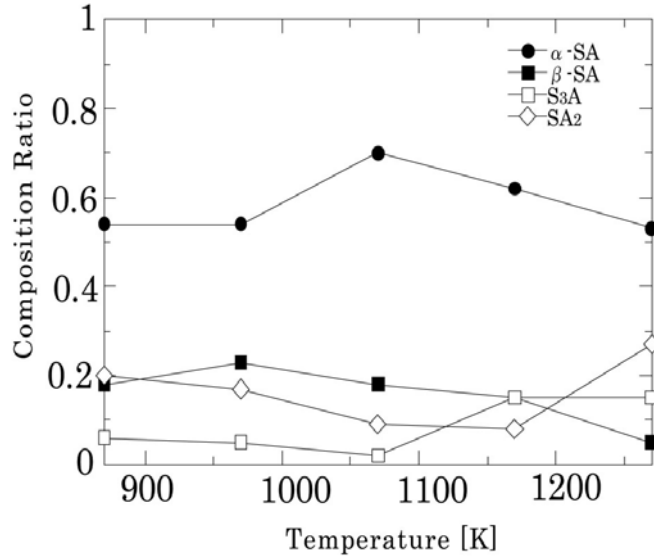

Fig. 4　Pre-heating effects on composition ratios
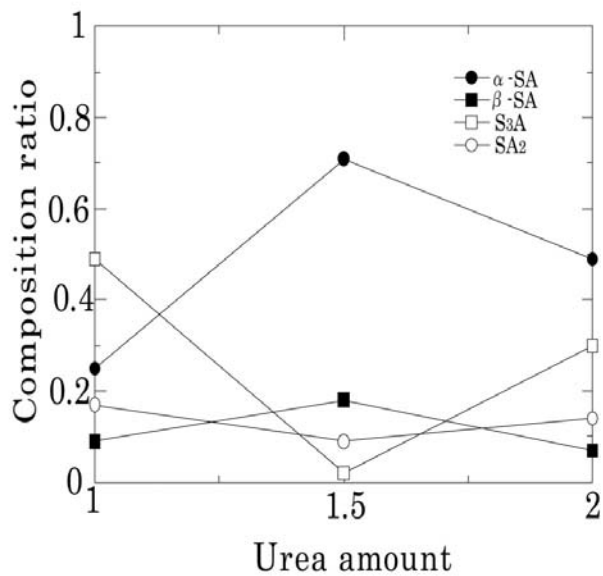(excess of urea: 50%)

Fig. 5　Excess urea effects on composition ratio
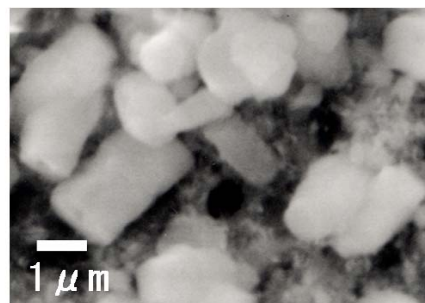(furnace temp.: 1070K)

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As a conclusion, strontium aluminates powders, which are well-known matrix materials in fluorescent applications, have been synthesized by a solution combustion synthesis technology with reactants of strontium nitrate, aluminum nitrate, and urea. The phase changes of synthesized $SrAl_2O_4$ structures have been investigated to make clear the effects of reactant concentrations and pre-heating temperatures of solution combustion synthesis.

The usual products of the reactions of fuels are CO_2, H_2O, and N_2, therefore, the elements of carbon and hydrogen are considered as reducing elements which correspond to valences of +4 and +1, respectively. Metal ions of Sr and Al are also considered as reducing elements in the process of combustion synthesis with the valences of +2 and +3, respectively. Total valences of the fuels with $CO(NH_2)_2$, $CO(N_2H_3)_2$, and $NH_2(CH_2)COOH$ are +6, +8 and +9, respectively. The ratios of the gas elements exhausted by combustion reactions with one mole of $CO(NH_2)_2$, $CO(N_2H_3)_2$, and $NH_2(CH_2)COOH$ are 4, 6 and 5, respectively.
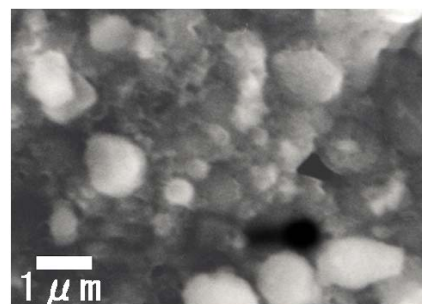
The ratio of a monoclinic phase compared to the hexagonal phase in the products of $SrAl_2O_4$ obtained by the present solution combustion synthesis has been studied by changing pre-heating temperatures and urea amount in order to varying calculated adiabatic temperature.

(a) 870K

(b) 1070K

Fig. 6   SEM images of the products

The present conditions were: the range of pre-heating temperature between 370K and 1270K, and the urea amounts 1.0, 1.5, and 2.0. Combustion synthesis reactions are occurred above 870K. With increasing pre-heating temperature from 870K to 1270K, the hexagonal-$SrAl_2O_4$ existed at whole temperature ranges, and the ratio of the monoclinic-$SrAl_2O_4$ became the maximum at 1070K. In addition, $Sr_3Al_2O_6$ and $SrAl_4O_7$ also existed.

With increasing pre-heating temperature the ratio of $Sr_3Al_2O_6$ increased and the ratio of $SrAl_4O_7$ decreased. With increasing urea amount from stoichiometric amounts, the ratio of monoclinic and that of hexagonal $SrAl_2O_4$ became the maximum and those of $Sr_3Al_2O_6$ and $SrAl_4O_7$ became the minimum at 1.5 times of stoichiometric urea amount. Phase composition of strontium aluminates is controlled with changing combustion synthesis conditions such as furnace temperature and urea ratio.

4. Nanometer-sized Long-duration Luminescent Materials

The principle of long-duration luminescent mechanism, which is shown in Fig. 7, can be considered as follows;

    (1) Lightening makes several electrons exited from valence band to conduction band,
    (2) Exited electrons are trapped just below the conduction band. With thermal fluctuation, the trapped electrons can reach at the luminescent center,
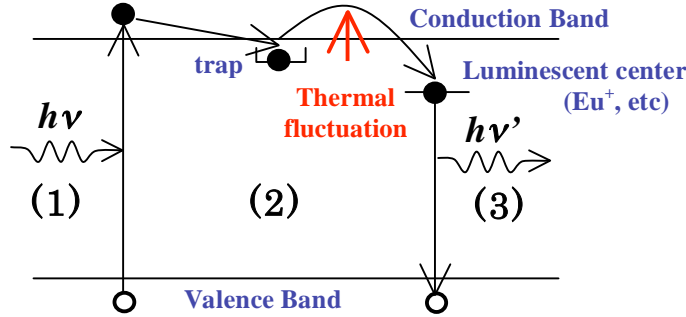   (3) By excited electrons move to the valence band, light is emitted.

Fig. 7   Typical model of long-duration luminescent process
(1): excitation by lightening, (2): electron trapped and activated to luminescent centers by thermal fluctuation, (3): luminescence with characteristic wavelengths

Figs. 8-(A) and 8-(B) show designs for longer-duration and stronger-luminescence with increasing the trap sites and luminescent centers, respectively. Selections of matrix structures and rare-earth additives play important roles to realize the designs. As a conventional recognition, the optimum size of luminescent powders has been considered to be around 20 mm, and with the decrease of size the luminescent power weaker. Recently, it has been made clear that the luminescent power becomes stronger more than 10 times compared to the case with the conventional optimum powder size when it becomes less than 10nm. Tables 1 and 2 show various useful luminescent materials with typical wavelength. Although the role of matrix structure on the increase of trap sites has not been cleared, the control of the matrix component bonding may work a key for achieving longer-duration.
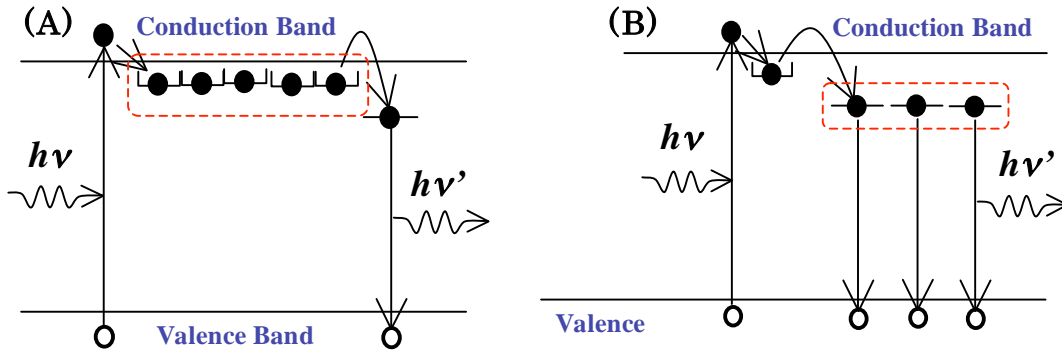
Fig. 8   Design for achieving long and bright luminescent materials
(A): for more long-duration, (B): for more bright-luminescence

Table 1   Typical luminescent materials

Matrix compound	Activated ion	Luminescent wavelength
$3Ca_3(PO_4)_2 \cdot Ca(F,Cl)_2$	Sb^{3+},Mn^{2+}	480, 585nm
$(Ba,Ca,Mg)_{10}(PO_4)_6Cl_2$	Eu^{2+}	483nm
$(Sr,Ca,Ba)_{10}(PO_4)_6Cl_2$	Eu^{2+}	447nm
$Ba_2P_2O_7$	Ti^{4+}	483nm
$(Sr,Mg)_3(PO_4)_2$	Sn^{2+}	620nm
$(Ca,Zn)_3(PO_4)_2$	Tl^+	328nm
$Sr_2P_2O_7$	Eu^{2+}	420nm
$LaPO_4$	Ce^{3+},Tb^{3+}	543nm
Zn_2SiO_4	Mn^{2+}	523nm
$(Sr,Ba)Al_2Si_2O_8$	Eu^{2+}	400nm
Y_2SiO_5	Ce^{3+},Tb^{3+}	530nm
$BaMg_2Al_{16}O_{27}$	Eu^{2+}	450nm
$Sr_4Al_{14}O_{25}$	Eu^{2+}	400, 490nm
$LiAlO_2$	Fe^{3+}	740nm
Y_2O_3	Eu^{3+}	610nm
YVO_4	Dy^{3+}	570nm

Table 2   Typical long-duration luminescent materials

Luminescent wavelength(nm)	Synthesis Method	$SrO:Al_2O_3$	Literatures
519	Sol-gel	1:1	J. Phys. Chem, B107 (2003) 3991
520	Solid-state	1:1	J. Ceram. Soc Jpa, 104 (1996) 322
(475)	Solid-state	1:2 (5:4)	J. Alloys Comp., 348 (2003) 224
(520)	Solid-state	1:1	J. Euro. Ceram. Soc, 20 (2000) 2129
(480)	Solid-state	4:7	Mat.Lett, 51 (2001) 14
(520)	Solid-state	1:1	J. Electrochem. Soc, 143 (1996) 2670
(513)	Combustion	1:1	Mat.Lett, 58 (2004) 352
(490)	halide	1:1	Mat.Lett, 54 (2002) 70
515	Combustion	1:1	Mat.Lett, 58 (2004) 1087
488	Solid-state	4:7 (1:1)	Mat.Scie. Eng, B57 (1998) 18
516	Solid-state	1:1	J. Lumi, 97 (2002) 1
	Soft-chemical	1:1	Mat.Chem. Phys, 65 (2000) 103
(500)	Sol-gel	1:1	J. Euro. Ceram. Soc, 20 (2000) 2129

5. Process design of nanometer-sized combustion synthesis

Fig. 9 shows a concept design of an apparatus for a new solution combustion synthesis technology, which has been identified and defined from the idea of conventional emulsion and solution combustion synthesis.   Emulsion combustion technologies have been already developed in industrial applications, which have been admired in general as the one superior to energy saving and combustion-pollution reduction.   However, the control of its stable combustion process is rather in problems because of controlling difficulties of its systematic pressure fluctuation during combustion propagation.   An accident caused from hydrogen-explosion through emulsion combustion happened in November, 2005 has taught us the importance to study on precise partial pressure and temperature distributions in the system for reducing their mismatching.
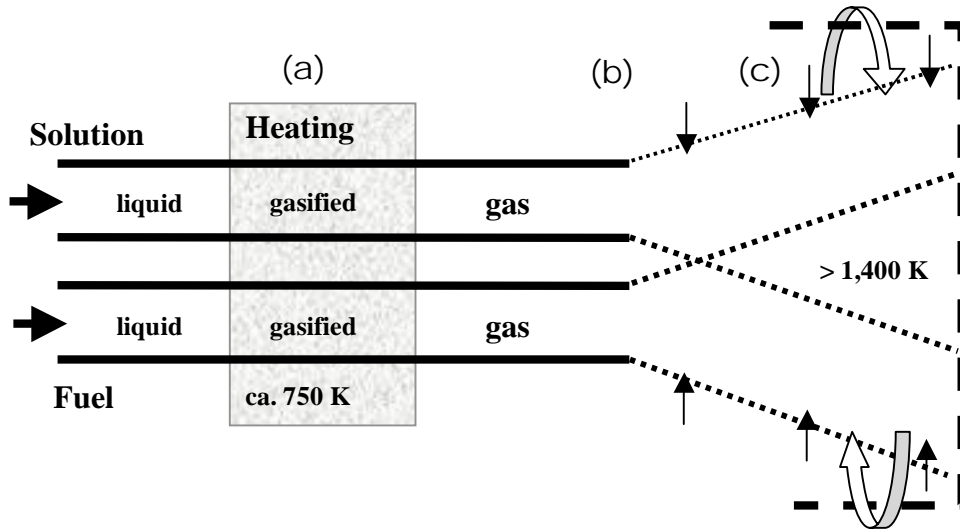

Fig. 9　　Concept design of a new solution combustion synthesis technology
(a): self-sustaining heating zone
(b): nozzles for accelerating and mixing reactant and solution lines
(c): combustion plane-zone assisted by rotating the combustion front wall

The characteristic merits of the present method are in (A) self-sustaining evaporation system assisted with combustion heat and (B) wall rotation system of reactant flow lines for uniform and stable reaction performance.   Since the nozzles are open to the atmospheric pressure before the combustion front, the process can be controlled much in higher reliability compared to conventional emulsion and liquid spray technologies. The volume of evaporated gas by self-sustaining heating, Vo in m^3/s, is estimated as equation (3);

$$Vo = \frac{Vi}{60 \cdot m} \cdot a \cdot \frac{T + 273}{273} \qquad (3),$$

where Vi is the liquid volume in cm^3/min, m molecular weights of solution and fuel, a gas molar volume in m^3 and T the combustion temperature in degree C.   The gas velocity, s in m/s, is calculated as the following equation (4) with the data of Vo and the tube diameter d in mm;

$$s = \frac{2 \times 10^6 \cdot Vo}{\pi \cdot d} \qquad (4)$$

By substituting 10, 18 for water/170 for lamp oil, 22.4×10^{-3}, 1500 and 2, for Vi, m, a, T and d, respectively, s of water and oil are determined to be 429 m/s and 45 m/s from equations (3) and (4).

The flow pattern of gas can be designed in Fig. 10 under the consideration of gas diffusion, D, as $D \propto x$ and $D \cdot s_0 = const.$: linear dispersion of gas in the direction of flow with gases around, which makes the gas velocity decreases with increasing the dispersing diameters. Then, the following relationship is also estimated, which can guide for the scale-up design;

$$s \propto \frac{1}{\sqrt{x}} f\left(\frac{y}{x}\right) \qquad (5)$$
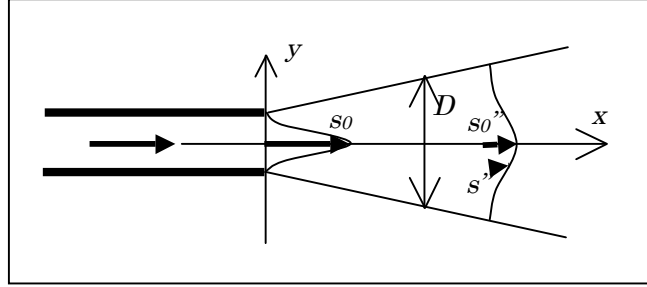

Fig. 10   Flow patterns of mist dispersion

In the present study, the emulsion combustion technology is combined with a mist combustion process. Since the combustion process is assisted with steamed water generated by heating with the reactant heat during sustaining combustion, fuels such as oil can completely burn without any polluted soot.   This technology can attain almost 50% energy saving with water assistance and form finer product powders through the process.   With the present prototype apparatus shown in Fig. 11, the effects of water addition could be obtained as the data of temperature changes in combustion flame as shown in Table 3.
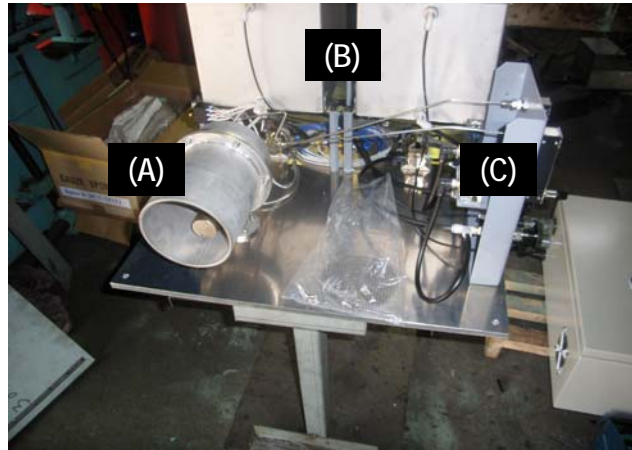

Fig. 11   Prototype apparatus for new solution combustion synthesis
(A): combustion chamber
(B): solution and fuel tanks
(C): flow controller

The comparison tests have been carried out for the cases of lamp and fuel oils.   In the case of lamp oil combustion, the temperature decreases with increasing water addition. On the other hand, the compatibility of A-fuel oil and water seems to be effective until 50% addition of water without any meaningful decrease of temperature.

Table 3   Combustion temperature changes during the oil/water mist combustion

Fuel(100cc/min)	Combustion temperature (K) with water addition				
	20 cc/min	30 cc/min	40 cc/min	50 cc/min	60 cc/min
lamp oil	1150	1120	1010	1020	980
A-fuel oil	1550	1550	1550	1500	1420

The present process can be controlled as follows;

(1) to heat the lines of reactant solution(water, etc.) and fuel liquid(oil, etc.) which are pumped and flown through the separated tubes,

(2) to ignite the steam-mixture of fuel and solution formed during their flow in a proper-sized pipe after flowing through nozzles,

(3) to control the amounts of solution and fuel evaporated with the self-sustaining heating for attaining complete combustion, and

(4) to control the reactant stable mixture flow and combustion propagation with proper rotations of fins set inside the tube.

The control parameters of the process are under investigation for applying to the present solution combustion synthesis technology.   Since the natural convection can be controlled by rotations of walls, the mixture flow is expected to be more uniform and stable as shown in Fig. 12.   Rotating gas evaporation is a new technique for continuous reduction of thermal convection using centrifugal and Coriolis forces.   The part of the combustion front has been made of meshed copper plate, which has been corroded with
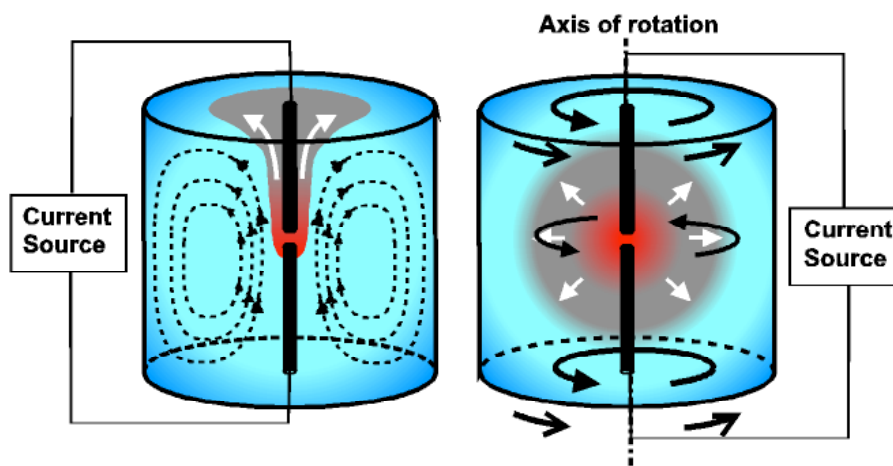

Fig. 12   Rotating gas evaporation using centrifugal and Coriolis forces

activated steam water.   The apparatus materials are now designed with non-corrosive and thermally-stable materials such as Si_3N_4.

6. Conclusion

This new technology has been originally proposed through the combinational technologies of solution and/or emulsion combustion synthesis, which will perform not only fine powders production but also highly efficient resource utilization with the aid of water, resulting in somewhat system-pressure-controlled energy saving before/after combustion propagation.   This proposed technology will be finalized in the final report of the present program by establishing the parameters for optimum machine design. The optimum machine will be successively completed during a new program.